MS44 O4

Determination of Fast and Irreversible Phenomena by Time-Resolved Diffraction. Caroline Curfs[a], Ann Terry[b], Miguel-Angel Rodriguez[c], Erich Kisi[d], Gavin Vaughan[a], Åke Kvick[a]. [a]*ESRF, Grenoble France.* [b]*ISIS, Didcot, UK.* [c]*ICV, Madrid, Spain.* [d]*The University of Newcastle, Newcastle, Australia.* E-mail: curfs@esrf.fr

Keywords: time-resolved diffraction, reaction mechanisms, composites

Irreversible phenomena can be extremely difficult to study, especially when the succession of events is extremely fast. Thus, in order to avoid missing information, the phenomenon has to be probed in the most continuous way.
Combustion synthesis [1] is a good example of fast and irreversible phenomenon. This synthesis is based on the exothermic properties of the chemical reaction to synthesize a whole sample. Two combustion modes exist depending whether the synthesis occurs in the form of a wave traveling through the sample (Self-propagating High-Temperature Synthesis SHS) or if it occurs simultaneously in the whole sample (Explosive mode). It can take only few seconds to obtain the final products and the lifetime of some intermediate phases can be as short as a couple of hundreds of milliseconds.
The technique traditionally used to determine the synthesis mechanisms, is based on the microscopic analysis of a sample in which the propagation of the front has been quenched. However, with this technique, no information on the time scale is available and some artifacts due to the quenching can be introduced. Moreover, when the system studied is complicated and composed of a large number of elements, the series of events happening during the synthesis can be impossible to determine. Thus, only in-situ studies can probe the reaction with enough details to enable the determination of the synthesis mechanisms. Time-resolved diffraction using synchrotron X-rays (TRXRD) [2] and Neutron (TRND) [3] can be used to determine, as the reaction proceeds, the sequence of appearance and disappearance of the crystalline phases as a function of time. These data, correlated with a study of the temperature evolution, give enough information to reconstruct a possible synthesis mechanism.
To illustrate these techniques, the combustion synthesis of an equiatomic mixture of Aluminium, Nickel, Titanium and Carbon (graphite) has been studied ex-situ by the "quenching" technique and in-situ by time-resolved diffraction. Synchrotron X-rays were used to study the self-propagating mode and neutrons the thermal explosion mode. TRXRD were performed on ID11. ESRF, France using the high-speed CCD camera FreloN. A 2D diffraction pattern can be taken every 135 ms, including 25 ms of exposure, and a 1D pattern in a quite continuous way (less than 1 ms between 2 patterns). The TRND were performed on the D20 instrument at ILL, France, with a 1 dimensional pattern every 500 ms (100 ms between 2 patterns. The results obtained have shown that with the same initial samples (composition and density), the products obtained were identical but the synthesis mechanisms were different depending on the combustion modes. For the SHS mode, 3 intermediate phases were observed and none for the TES. Moreover the triggers for the reaction were different: NiAl for the SHS and TiC for the TES.

[1] Merzhanov A.G., *Combustion and Plasma Synthesis of High-Temperature Materials*, VCH, New York, 1990, 1.
[2] Curfs C., Terry A.E., Vaughan G.B.M., Turrillas X., Rodriguez M.A., Kvick Å., *J. Eur. Ceram. Soc*. 2002, 22, 987. [3] Riley D.P., Kisi E.H., Hansen T.C., Hewat A.W., *J. Am. Ceram. Soc.* 2002, 85, 2417.

MS44 O5

The PILATUS 6M Detector: A Powerful Instrument for Advanced Diffraction Studies Miroslav Kobas, Christian Broennimann, *SLS Detector Group, Swiss Light Source, Paul Scherrer Institut, CH-5232 Villigen PSI, Switzerland.*
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Keywords: X-ray detector technology, detector development, protein crystallography

A novel x-ray detector has been developed at the Paul Scherrer Institut for protein crystallography at the Swiss Light Source (SLS). The PILATUS 6M detector is a two-dimensional hybrid pixel array detector, which operates in single-photon counting mode [1]. It consists of 2527 x 2463 pixels with a pixel size of 0.172 mm.
This detector features several advantages compared to current state-of-the-art CCD and imaging plate detectors. The main features include: no readout noise, superior signal-to-noise ratio, a point spread function of one pixel, readout time of 5 ms, framing time of 100 ms, a dynamic range of 20bit, high detective quantum efficiency (100% at 8 keV, 80% at 12 keV, 50% at 16 keV) and the possibility to suppress fluorescence by a energy threshold. The short readout and fast framing time allow to take diffraction data in fine-phi-slicing mode with continuous rotation of the sample without opening and closing the shutter for each frame.
Because of the specified properties, this detector is especially suited for the study of weak diffraction phenomena, time-resolved experiments, accurate measurements of Bragg intensities and resonant scattering experiments.
Results from various x-ray experiments are presented, including diffraction data from protein and aperiodic crystals, as well as results from diffuse scattering and x-ray absorption experiments. All data have been collected with the PILATUS 6M detector at the X06SA beamline of the SLS.

[1] Broennimann, Ch. et al., *J. Synchrotron Rad.*, 2006, 13, 120-130.